SPL TR 69-3

ELECTRON MULTIPLIER DEVELOPMENT (PHASE I)

by

Wan Sang Chung
Chih-shun Lu

January 1969

Prepared under
Contract NSR 33-144-003

by

Syracuse University Research Corporation
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ELECTRON MULTIPLIER DEVELOPMENT (PHASE I)

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SUMMARY

The feasibility of fabricating a thin film window to permit the transmission of primary electrons of energy range 5-15 Kev but strong enough to sustain a pressure difference of one atmosphere was examined. The correlations between the electron transmission coefficient and the mechanical strength of thin films were studied both theoretically and experimentally. It was found that a composite film of aluminum and aluminum oxide with a total thickness of about 2000 Å and supported by a 75-mesh grid could meet the requirements. Techniques for preparing such a window are presented. Experimental results obtained from several films were satisfactory although further optimization and improvements are needed. Recommendations are made for further developmental effort directed toward the final realization of a practical sealed-off capillary-type electron multiplier.

1. INTRODUCTION

The objective of this program was to investigate the feasibility of preparing a thin film window for the capillary-type electron multiplier. The window should permit the transmission of energetic electrons but sustain a pressure difference of one atmosphere. Theoretical analyses indicated that such a window could be constructed. The main task was to fabricate a self-supported thin aluminum film about 2000 Å thick mounted on an appropriate window frame. The aluminum film was then anodized with part of the aluminum being converted to aluminum oxide in order to increase the mechanical strength. The back surface of the thin film window can be coated with a high secondary electron emissive material, such as a layer of porous KCl, to enhance the overall electron yield. The following sections describe the detailed techniques of fabricating a thin film window to be used specifically for a sealed-off capillary-type electron multiplier.

2. THEORETICAL ANALYSIS

2.1. Transmission of Electrons through Thin Films

In order to obtain the basic parameters for the design of a thin film window which meets all the requirements, the range-energy relation of electrons transmitted through a thin film will be discussed. This information is necessary for the determination of proper thickness of the film to be used as a window. One of the basic requirements of the window is to have sufficient transmittance for electrons in the energy range of 5 to 15 Kev.

Average energy loss per unit path length for nonrelativistic electrons in the unit of erg-cm^{-1} is given by the expression (Ref. 1),

$$-\frac{dE}{dx} = \frac{4\pi N q^4}{mv^2} \ln\left[\frac{mv^2}{2\bar{I}} \left(\frac{e}{2}\right)\right]^{1/2}, \qquad (2\text{-}1)$$

where N is the number of electrons per cm^3, $\bar{I}$ is the average excitation potential of the atoms of the film material in ergs, and e = 2.718. The charge, rest mass, and velocity of the electron are represented by q, m, and v in the cgs unit system, respectively.

Equation (2-1) may be expressed in the unit of Kev per (mg/cm^2) by the expression,

$$-\frac{dE}{dX} = 0.307 \frac{Z}{A} \frac{1}{\beta^2} \left[\ln(51.1\beta^2) - \ln\bar{I} + 8.67 \right], \qquad (2\text{-}2)$$

where β is the ratio of velocity of the incident electrons to the velocity of light in vacuum, and $\frac{Z}{A}$ the ratio of atomic number to mass number of the film material. Here we introduced $X = \rho x$, where ρ is the density of the material in g/cm^3. The average excitation potential $\bar{I}$ in (2-2) is represented in the unit of ev.

Although the proposed window consisted of a composite film of aluminum and aluminum oxide, for the sake of simplicity only the transmission of electrons through a thin aluminum oxide film will be calculated. If one neglects the chemical bonding, the effective values of $\frac{Z}{A}$ and $\bar{I}$ for a material which is represented by the chemical formula $(Z_1)_{n_1}(Z_2)_{n_2}$ can be expressed by the relations,

$$\left(\frac{Z}{A}\right)_{eff} = \frac{n_1 Z_1 + n_2 Z_2}{n_1 A_1 + n_2 A_2}, \tag{2-3}$$

and

$$\ln \bar{I}_{eff} = \frac{n_1 Z_1 \ln \bar{I}_1 + n_2 Z_2 \ln \bar{I}_2}{n_1 A_1 + n_2 A_2}. \tag{2-4}$$

If the approximate expression for the average ionization potential

$$\bar{I}(Z) \approx 13.5\, Z \text{ (ev)} \tag{2-5}$$

is used(Ref. 2), one obtains $\ln \bar{I}_{eff} = 2.425$ and $\left(\frac{Z}{A}\right)_{eff} = 0.490$ for aluminum oxide. By substituting these values into (2-2), the average energy loss for incident electrons with different energies may be calculated. The mean range can then be obtained from the equation,

$$R = \int_{\epsilon}^{E'} \left(\frac{dE}{dX}\right)^{-1} dE, \tag{2-6}$$

where ϵ is the energy below which the logarithm in (2-1) becomes negative, and E' is the incident energy of electrons. The integral in (2-6) can be evaluated graphically as the area under the curve $\left(\frac{dE}{dX}\right)^{-1}$ versus E.

Using a density of 2.8 gm/cm^3 for the amorphous aluminum oxide film (Ref. 3), the inverse stopping power and the range for electrons with incident energy ranging from 1 to 15 Kev were calculated. Table 1 shows the results of such calculations. The range versus the incident energy of electrons for the aluminum oxide films is shown in Figure 1. These values are in good agreement with the experimental results on the penetration powers of primary electrons through Al_2O_3 films reported by Stroh and Welsch (Ref. 4).

Based on the above calculations, the film thickness has to be about 2000 Å in order to have reasonable transmission for electrons with energy as low as 5 Kev.

2.2. Calculation of the Mechanical Strength of Thin Films

Because the window has to satisfy the requirement of sustaining a pressure difference of one atmosphere, the mechanical properties of a thin film will be briefly discussed. It is well known that the properties of thin films, particularly the mechanical properties, are highly sensitive to the film preparation techniques and can be drastically different from the bulk properties. Thus the correlation between the experimental results and a highly idealized theoretical model is often very difficult and even meaningless. However, qualitative information can still be inferred from the basic stress analysis of considering the film as a thin and uniform membrane.

For a circular membrane with the edge clamped and under uniform load, the maximum stress is at the edge of the membrane and is given by the relation (Ref. 5),

$$S = \frac{3}{4}\frac{wr^2}{t^2}, \qquad (2\text{-}7)$$

where S is the maximum stress in dynes/cm^2, w is the load per unit area in dynes/cm^2, r is the radius of the membrane in cm, and t is the thickness of the membrane in cm.

Using the average value of 1.74×10^9 dynes/cm^2 for the fracture strength of aluminum oxide films obtained by Grosskrentz (Ref. 6), the minimum thickness for a film with 0.1 cm radius to support a pressure difference of one atmosphere is about 2×10^5 Å. The opening at the front end of a capillary-type electron multiplier is about 0.1 cm in radius. If this opening is to be covered by a thin film window to sustain the pressure difference of one atmosphere, the window will be too thick for the transmission of electrons. Therefore, a fine grid structure is apparently needed to give additional support to the film.

3. EXPERIMENTAL RESULTS

3.1. Sample Preparation

3.1.1. Film Holders. -- A film holder has to meet the dual functions of mechanical support and electrical connection to the thin film window. A typical film holder is shown in Figure 2. The holder was made of aluminum. A nickel grid (75-mesh with 77% open area) of 3.05 mm in diameter was placed on the tapered edge of the holder. The objective of adding a grid to support the film was to increase the mechanical strength of the film as discussed in the last section.

3.1.2. Preparation of Collodion Films. -- In choosing a supporting film for aluminum deposition, certain requirements must be satisfied. The supporting film should be easily formed. It must be of uniform thickness without wrinkles or defects, and should be capable of being readily transferred and mounted on the film holder. In addition to the mechanical, thermal, and chemical resistances required by the supporting film during the aluminum film deposition process, it must be dissolvable once the aluminum film is formed. In the early stage of this program the collodion film appeared to fulfill all of these requirements. The fabrication procedures for the collodion films were as follows:

1. A few drops of collodion solution (10% collodion in amyl acetate by weight) were dropped on the water surface to form a thin layer.

2. After a few seconds when the collodion layer was partially condensed, the water level was then lowered so that the collodion film just touched the tapered edge of the film holder.

3. After the collodion film was completely condensed, it was cut around the edge of the film holder using a sharp blade. The film holder which had the collodion film firmly attached was then lifted from the water and transferred to under an infra-red lamp (250W) for about half an hour to remove excess water.

Figure 3 illustrates the procedures described above. A water surface apparatus (manufactured by Ernest F. Fullam, Inc., Schenectady, New York) was used to control the water level. Since it was found that the defects formed in the films were mainly due to the dust particles and impurities during the preparation processes, it was important to use distilled water and to carry out the above processes in a laminar-flow working station. Extreme precautions were taken to avoid dust contamination during transfer of the films to and from the vacuum system. A microscope with magnification up to 400X was used to examine the film imperfections.

3.1.3. Preparation of Aluminum Films. -- Aluminum film was deposited in a conventional vacuum system utilizing a 6-inch oil diffusion pump to evacuate an 18-inch bell jar. High purity (99.99%) aluminum in wire form was evaporated from a tungsten coil located at a distance of about 28 cm below the film holder. The pressure during evaporation was about 2×10^{-7} Torr. A quartz crystal oscillator unit was used to monitor the thickness during evaporation. The actual thickness of the aluminum film was measured by the interferometric method on a separate thickness sample deposited on glass substrate which was located adjacent to the film holder during the evaporation.

A series of aluminum films with thicknesses from 1,000 to 2,500 Å has been prepared. It was found that for thicker aluminum coatings on the collodion films, deformation was often observed. This appeared to be due to the heating effect on the collodion film during evaporation of the aluminum and the mechanical stress introduced by the aluminum film. To solve this deformation problem the evaporation rate was limited to about 80 to 120 Å/sec. Inside the bell jar, a shutter was used to cover the evaporant during preliminary

outgassing and thus minimizing the preheating effects on the collodion film. In this manner, aluminum films of thickness up to 2,500 Å were deposited on the collodion films without any deformation.

Typical photomicrographs of an aluminum film deposited on a collodion film backed by a nickel grid are shown in Figure 4.

3.1.4. Preparation of Aluminum Oxide Films. -- The aluminum oxide film was formed by the anodization of the aluminum film in an electrolyte. The electrolyte used was a 3% solution of ammonium citrate by weight in distilled water. Ammonium hydroxide was added to the electrolyte to establish a pH value of about 5.5. The anodization set up is shown in Figure 5. The anodization current was kept at about 0.6 mA, and the applied voltage was slowly increased. The anodized films were then cleaned with distilled water. Thicknesses of the aluminum oxide ranging from 600 to 800 Å were determined by the applied voltage through the thickness-voltage relation of 13.7 Å/volt (Ref. 7). The thickness of anodized film was limited by the microscopic break-down in the oxide film which caused erratic current flow. Such break-down is believed to be related to the defects in the aluminum films. Surfaces of the oxide films formed on aluminum specimens were microscopically smooth except for a few minor defects. The aluminum oxide film formed by this technique is known to be in amorphous form.

3.1.5. Removal of Collodion Films. -- After the formation of the anodized aluminum oxide film, the film was firmly sealed onto the film holder by water glass. The film holder was then placed in a vapor degreaser with amyl acetate vapor to dissolve the collodion film. Although there were no visible residues of collodion left after about 30 minutes of vapor cleaning, it was discovered later that the collodion between the aluminum film and the film holder can not be completely dissolved. When the film holder, which had the film attached, was heated in vacuum, the concealed collodion spread out and changed the characteristics of the aluminum film. It has been reported that collodion films can also be removed by heating the composited film structure in air at an elevated temperature (Ref. 8). However, such techniques were not tested in our laboratory, and instead, we adopted a different method of preparing the self-supported aluminum film to avoid the problem of removing the collodion film.

3.1.6. Alternate method for the Preparation of Self-Supported Aluminum Films. -- In order to eliminate the problem of residual collodion, an alternate method of preparing a self-supported aluminum film was used during the later stage of this program. This technique was a modification of a method originally developed by Vouros et. al. (Ref. 9).

Several drops of a 5% polyvinyl pyrolidone (PVP) in ethanol solution were applied on a reclined microscope slide to form a uniform layer. After the evaporation of ethanol the glass slide was covered with a uniform layer of PVP. Aluminum evaporation was carried out in a manner described in Section 3.1.3. A mask was used to produce films in the shape of a circular disk with a diameter of about 0.5 cm. The glass slide was then placed in a dish and distilled water was added until the water level touched the PVP. The PVP layer was slowly disolved and the aluminum films were left floating on the water surface. The water level was then increased so that a film holder with the nickel screen attached could be placed under the floating aluminum film. After careful withdrawal of the water, the aluminum film was brought down to settle on the film holder similar to the way that collodion film was prepared as described in Section 3.1.2.

The film holder which was described in Section 3.1.1. was also used in this technique although it was not originally designed for this particular technique. It appeared that a flat film holder would be more appropriate for this process but due to time limitation it was not able to optimize the design of film holders for this process.

3.1.7. Preparation of Potassium Chloride Layer. -- Among many secondary electron emitting materials, a low-density potassium chloride has received particular attention for its attractive features of high yield characteristics and simplicity of preparation(Ref. 10).

The potassium chloride evaporations were carried out in a shielding box inside a bell jar to prevent serious contamination of the vacuum system. The film holder with the composite Al_2O_3-Al film was placed at a distance of about 5 cm from a tantalum boat which contained 25 mg of KCl in powdered form. The system was first evacuated to a pressure of the order of 10^{-7} Torr. Dry

argon gas was then introduced continuously into the vacuum chamber through a leak valve. The pressure was maintained at about 2 Torr by using the mechanical pump only. The tantalum boat was then heated until the KCl was completely evaporated. In this manner, a porous KCl layer (estimated densities are between 1 to 5% of the normal density) of several tens of microns in thickness was prepared. A photomicrograph of the porous KCl film is shown in Figure 6. The thickness of the potassium chloride film was measured with an optical microscope, focusing in turn on the top of the film and on the surface of the film substrate.

3.2. Pressure Tests

To test the mechanical strength of the film under a pressure differential, the composite Al_2O_3-Al film which was supported by the nickel grid and the film holder was mounted on the opening of a small test chamber. The chamber was connected to a high vacuum pumping system so that a pressure difference could be established across the film. The chamber could be closed after evacuation by a high vacuum valve. This test chamber is shown in Figure 7. The pressure in the chamber was measured by a Pirani gauge and an ionization gauge. Ultra-high vacuum components were used to construct the chamber in order to minimize the problem of leak and outgassing.

To measure the leak rate of the thin film window, the chamber was first pumped down with the opening blocked by a blank flange. An infra-red lamp was used to heat the chamber for outgassing. When the pressure inside the chamber reached about 10^{-6} Torr, the chamber was allowed to cool to room temperature and the high vacuum valve was closed. After closing the high vacuum valve, the pressure in the chamber was measured as a function of time. Because the ionization gauge behaved as a small ion pump, only the Pirani gauge was used during the pressure versus time measurements. In this manner, the leak and outgassing rate of the chamber itself were determined. The blank flange was then replaced with a flange which had the thin film window mounted on the opening and the same procedure was followed for the measurement of pressure versus time. The data are plotted in Figure 8. The film used in this measurement consisted of 680 Å of aluminum oxide and 1300 Å aluminum.

Films with thickness lower than this were not able to sustain the pressure tests.

Since the volume of the chamber V was known, the leak rate due to the thin film window Q could be calculated by the following relation

$$Q = V \left(\frac{dp}{dt}\right)_w - \left(\frac{dp}{dt}\right)_o , \qquad (3\text{-}1)$$

where $\left(\frac{dp}{dt}\right)_w$ and $\left(\frac{dp}{dt}\right)_o$ are the rates of pressure change in the chamber with the window and with the opening blocked respectively. From Figure 8, one obtains $\left(\frac{dp}{dt}\right)_w = 8.0 \times 10^{-6}$ Torr/sec and $\left(\frac{dp}{dt}\right)_o = 2.5 \times 10^{-7}$ Torr/sec. Substituting these values into (3-1) and using V = 196 cm^3, the leak rate for that particular window was determined to be 1.5×10^{-6} Torr-liter/sec.

3.3. Measurements of Electron Transmission

The experimental set-up for the measurement of electron transmission through the thin film window is shown in Figure 9. A modified electron gun made by Superior Electronics Corporation (Model SE-2B) was used as the monoenergetic electron source. The heater and cathode were removed and replaced by a tungsten filament. The electrons produced by the filament were accelerated through a series of focusing electrodes and left the final aperture at the ground potential which was about 1 kv positive with respect to the filament. The window F was mounted to cover a holder on the plate P which was coated with phosphorous material for the visual indication of the electron-beam spot size. A variable power supply provided the high voltage ranging from 1 to 9 kv to the plate P. The maximum accelerating potential was limited by arcing at the electrical feedthroughs. The distance between the plate P and the last aperture was about 13 cm. A Faraday cage C covered with teflon was placed under the plate P to collect the electrons. The cage was negatively biased at 67.5 volts with respect to the plate P so that low energy secondary electrons would not be collected. A movable deflector D made of a thin aluminum sheet at the ground potential was used to steer the position of the electron beam.

The entire set-up was placed inside the bell jar and evacuated by a conventional high vacuum system. A Keithley Model 601 electrometer modified to withstand the high floating potential was used to measure the currents from the plate or the cage.

To obtain the electron transmission data, the cage current with no film on the film holder had to be measured first. A film holder with the nickel grid only, was mounted on the plate P. After the bell jar was evacuated to about 10^{-5} Torr, the electron gun filament and focusing electrode voltages were turned on. Before actual readings were taken, the system was allowed to stabilize thermally for about 30 minutes. The plate voltage was then set at the maximum value of 9.0 kv. The electron beam was focused on the plate by adjusting R_2, such that the cross section of the beam covered an area slightly larger than the window opening. Once the focusing condition of the beam was set, it was kept unchanged throughout the measurements. The beam was next deflected away from the window opening by the deflector D and the beam intensity was adjusted by R_1 so that the current collected by the plate (I_p) was 1.0×10^{-7} Å. Some cage current (I_c) did flow when the beam was displaced from the window and this was mainly due to the noise and scattered electrons. The beam was then moved back to the window and the plate current and cage current were recorded as I_p' and I_c' respectively. Therefore, the actual current passing through the opening of the nickel grid (I_o) was given by the expression

$$I_o = I_p - I_p' = I_c' - I_c. \qquad (3\text{-}2)$$

The above measuring procedures were repeated for different plate voltages at 1 kv increments.

For the measurements of electron transmission through the film, a fabricated film (without the KCl layer) with film holder was mounted on the plate P and the same procedure repeated. The actual current passing through the film could be calculated and was designated on I_w. The transmission coefficient was computed from the relation

$$T = \frac{I_w}{I_o} \times 100\%. \qquad (3\text{-}3)$$

The transmission coefficients of several films with different thicknesses and preparation techniques are shown in Figure 10.

4. DISCUSSIONS

4.1. Pressure Tests

The leak rate represented by Figure 8 for a particular film which consisted of 680 Å of Al_2O_3 and 1300 Å of Al was by no means the minimum value obtainable for such a film. With further refinements in fabrication process and the design of the film holder, it is expected that the leak rate can be further reduced. Even with the reported leak rate, a small ion pump with a pumping speed of a few tenths of liters per second will be able to maintain a capillary-type electron multiplier housed in an envelope at the required operating pressure ($< 5 \times 10^{-4}$ Torr). Methods for the integration of the thin film window to a commercially available channel electron multiplier have been discussed in detail in a SURC proposal (Ref. 11).

4.2. Electron Transmission Tests

The electron transmission coefficient for an aluminum film as thin as 500 Å prepared by the method using an intermediate collodion film for supporting was exceedingly low as can be seen from Figure 10. Although there was no visible collodion residue left after the vapor cleaning process, a yellowish layer appeared after the aluminum film was exposed to the electron beam for several hours. This was probably due to the collodion, which was concealed between the aluminum film and the film holder and could not be dissolved during the vapor cleaning process, and which smeared out and decomposed when heated by the electron bombardment under vacuum. This implied that the vapor cleaning process to remove the collodion film was inadequate. Either a more efficient way of removing the collodion film or a different method of preparing the self-supported aluminum film had to be developed. We tried

the latter approach and films prepared by a floating process showed reasonable transmission coefficients for electrons although the values were still lower than those reported in Ref. 2. The deviations are believed to be partially due to the instrumentation since the present set-up for electron transmission measurements was less than ideal. Considerable difficulty was experienced during the measurements. Scattered electrons and electrical breakdown were major problems in the present set-up. Therefore, the data presented in Figure 10 were only of qualitative nature. A more elaborated set-up is necessary in order to obtain more meaningful data on the electron transmission coefficients of thin films.

Due to the difficulties experienced during the electron transmission tests, the secondary electron emission characteristics of the porous KCl layer were not examined. However, the preparation of porous KCl layers were successfully carried out and one should expect a yield for secondary electrons of more than 5 as reported by Goetze, et.al., (Ref. 10), for such a layer.

4.3. Conclusion

We have demonstrated the feasibility of fabricating a thin film window which consists of a partially anodized aluminum film supported by a fine grid for a sealed-off capillary-electron multiplier. Such a window can withstand a pressure difference of one atmosphere with relatively low leak rate and have reasonably high electron transmission coefficients. Results better than the presently reported ones can be expected with further refinements of the fabrication processes. However, improvements such as the design of the film holders and the sealing process of the film to the holder can be meaningful only if the window and the capillary electron multiplier tube is to be considered as a whole. A program for the integration of the thin film window to the commercially available channel electron multiplier was presented in a SURC proposal(Ref. 11). Because of the many new applications of such a sealed-off capillary-electron multiplier in scientific research, it will be highly desirable to extend the present program towards the realization of a prototype sealed-off capillary-electron multiplier.

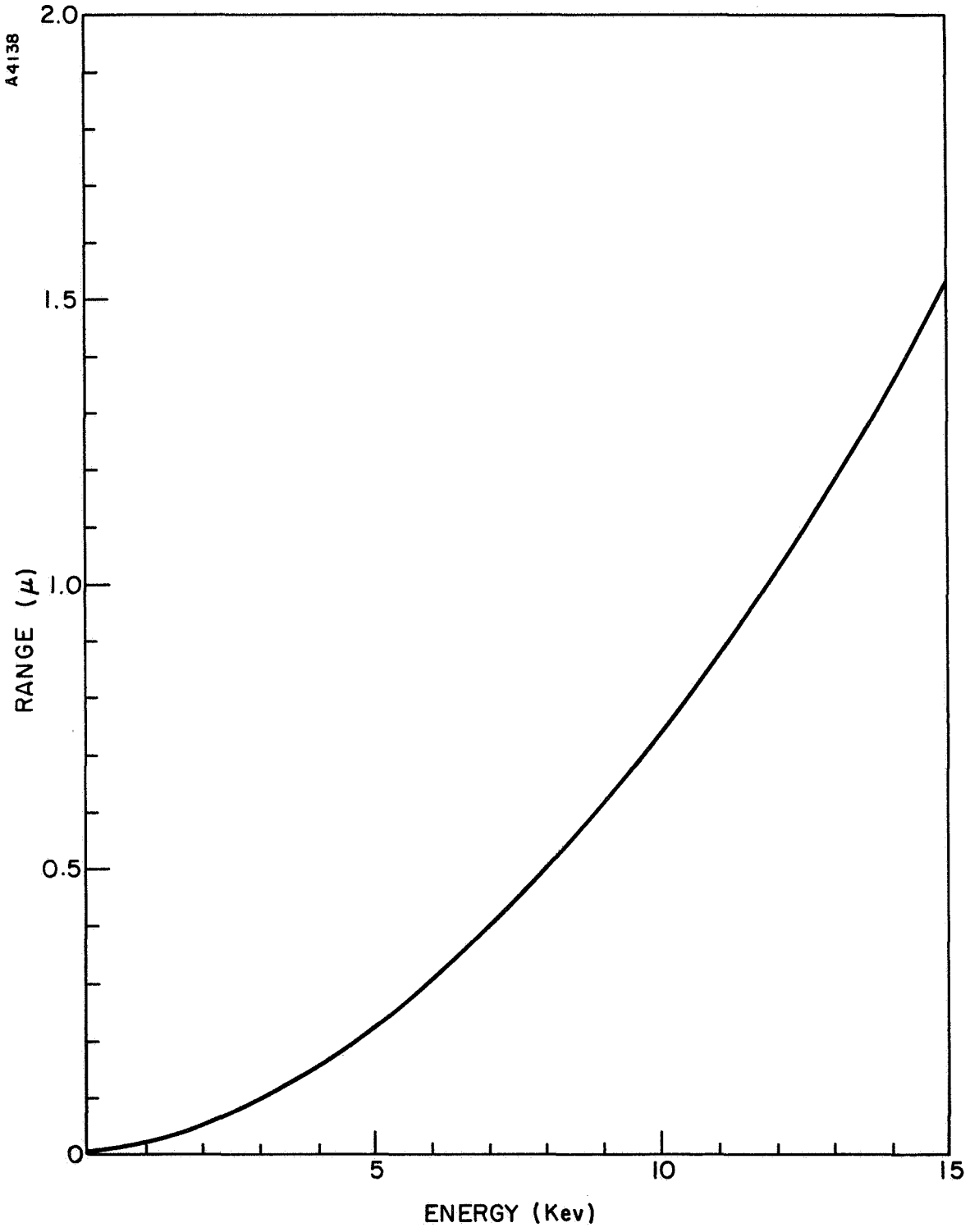


FIGURE 1. RANGE VERSUS INCIDENT ENERGY OF ELECTRONS IN ALUMINUM OXIDE FILM

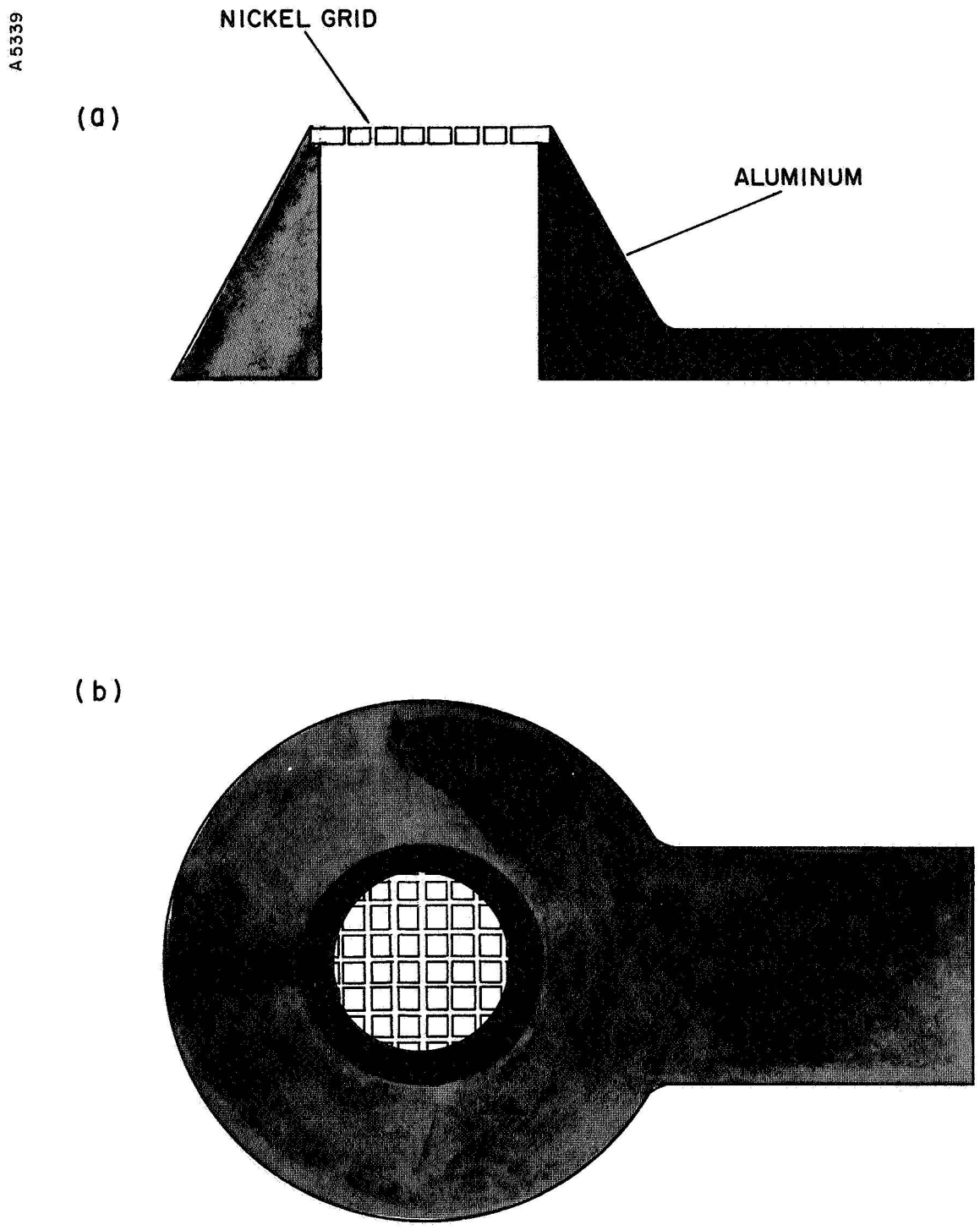
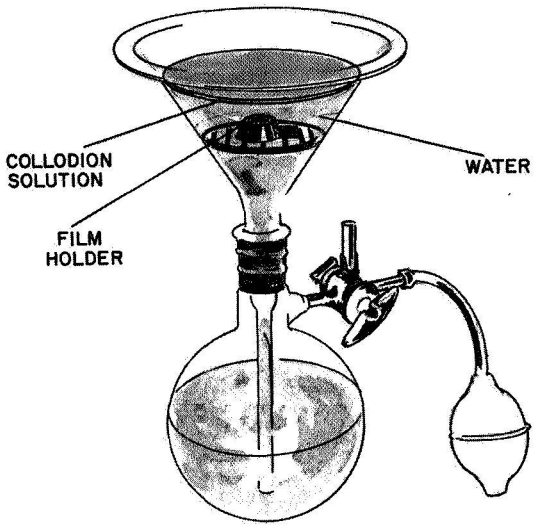
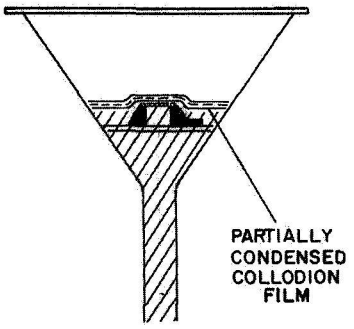


FIGURE 2. TWO VIEWS OF A FILM HOLDER WITH A NICKEL GRID

a. Collodion Solution Dropped on Water Surface to Form the Film

b. Water Level Was Lowered to Make the Collodion Film in Contact with the Holder

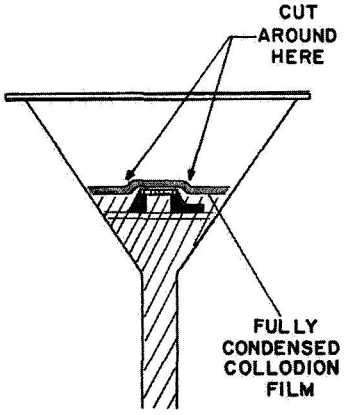




c. After Condensation

d. Collodion Film on Ni-Grid Sitting on Aluminum Holder

Note: Film Thickness not to scale



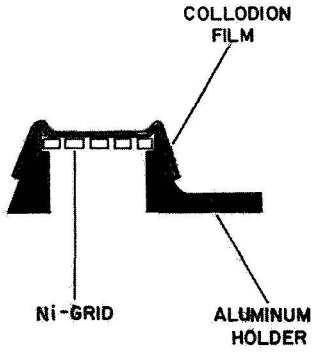
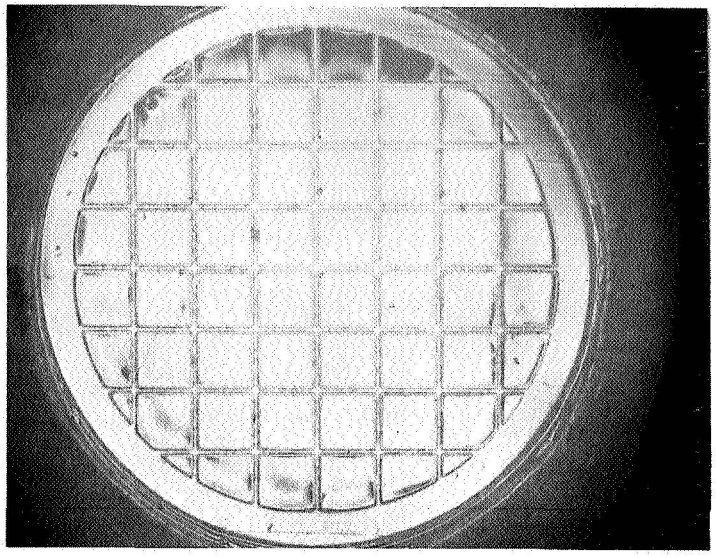
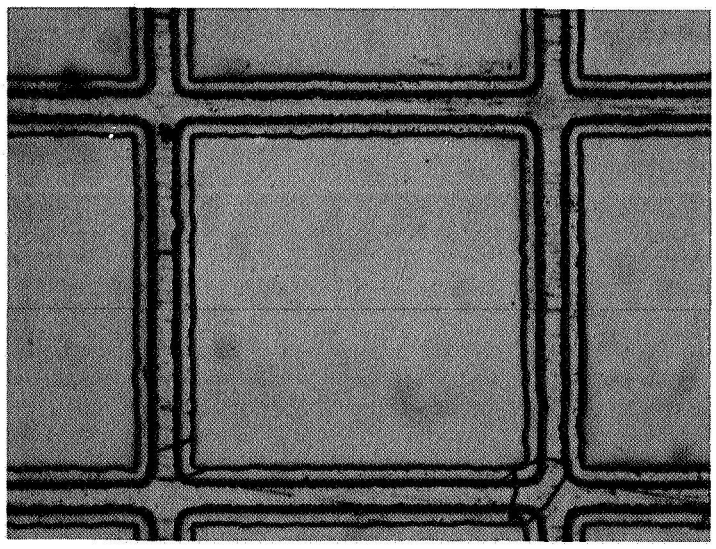


FIGURE 3. PROCEDURES FOR THE PREPARATION OF THIN COLLODION FILMS

A5337



a. Magnification 25x



b. Magnification 155x

FIGURE 4. PHOTOMICROGRAPHS OF AN ALUMINUM FILM DEPOSITED ON A COLLODION FILM BACKED BY A NICKEL GRID

A5338

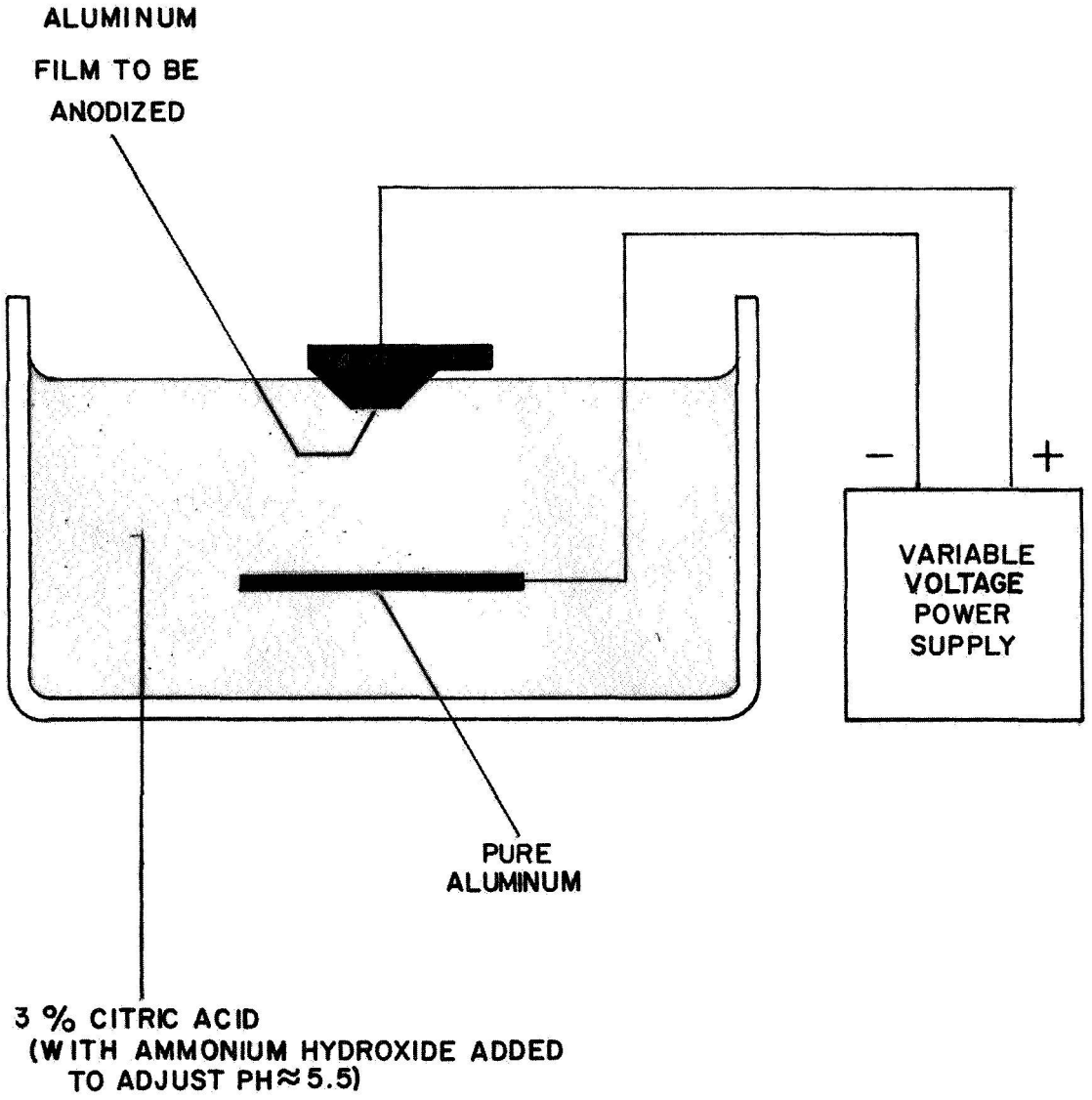


FIGURE 5. APPARATUS FOR THE ANODIZATION OF ALUMINUM FILMS

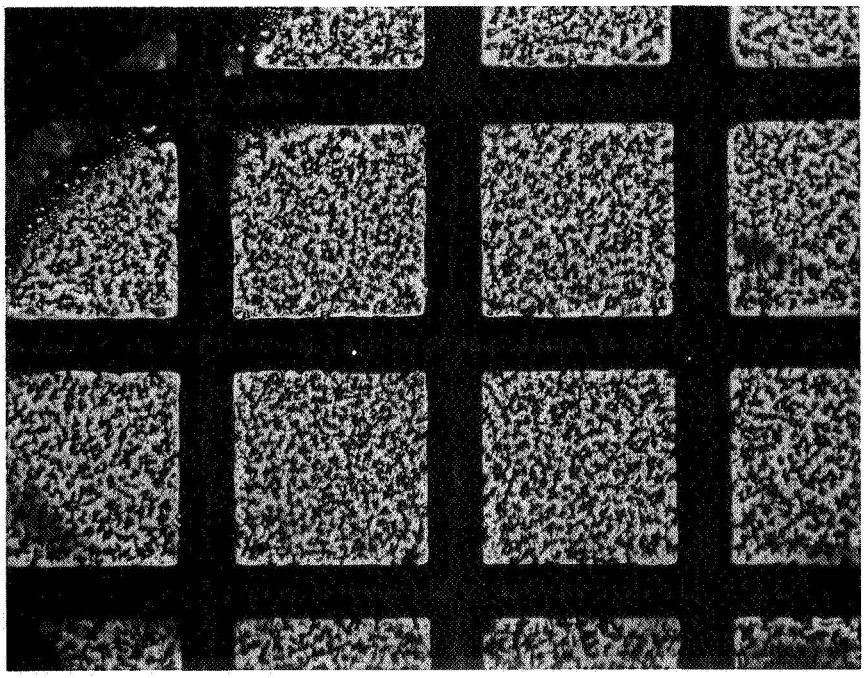
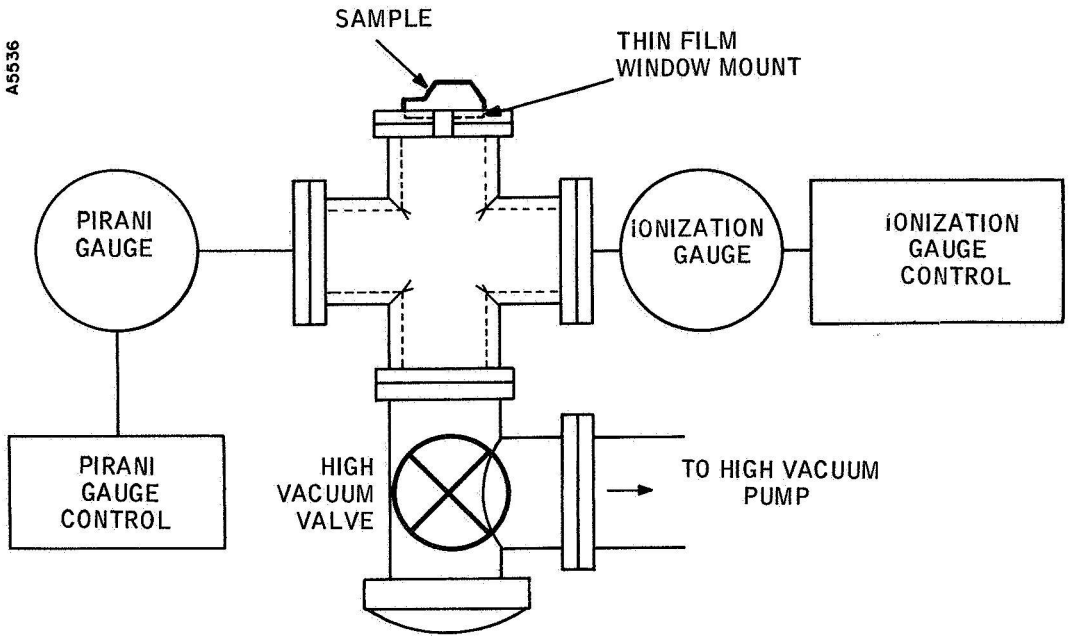
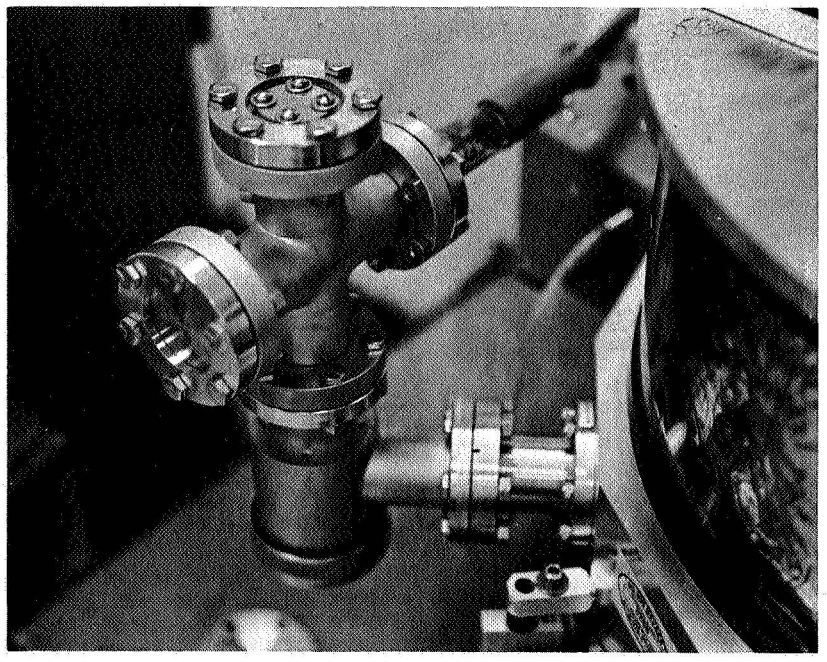


FIGURE 6. PHOTOMICROGRAPH OF THE POROUS KCl LAYER. MAGNIFICATION 100x.



a. Schematic Diagram for the Test Set Up



b. Pressure Test Chamber (Without the Sample)
(Pirani Gauge Not Shown)

FIGURE 7. PRESSURE TEST SET UP FOR THE FABRICATED FILMS

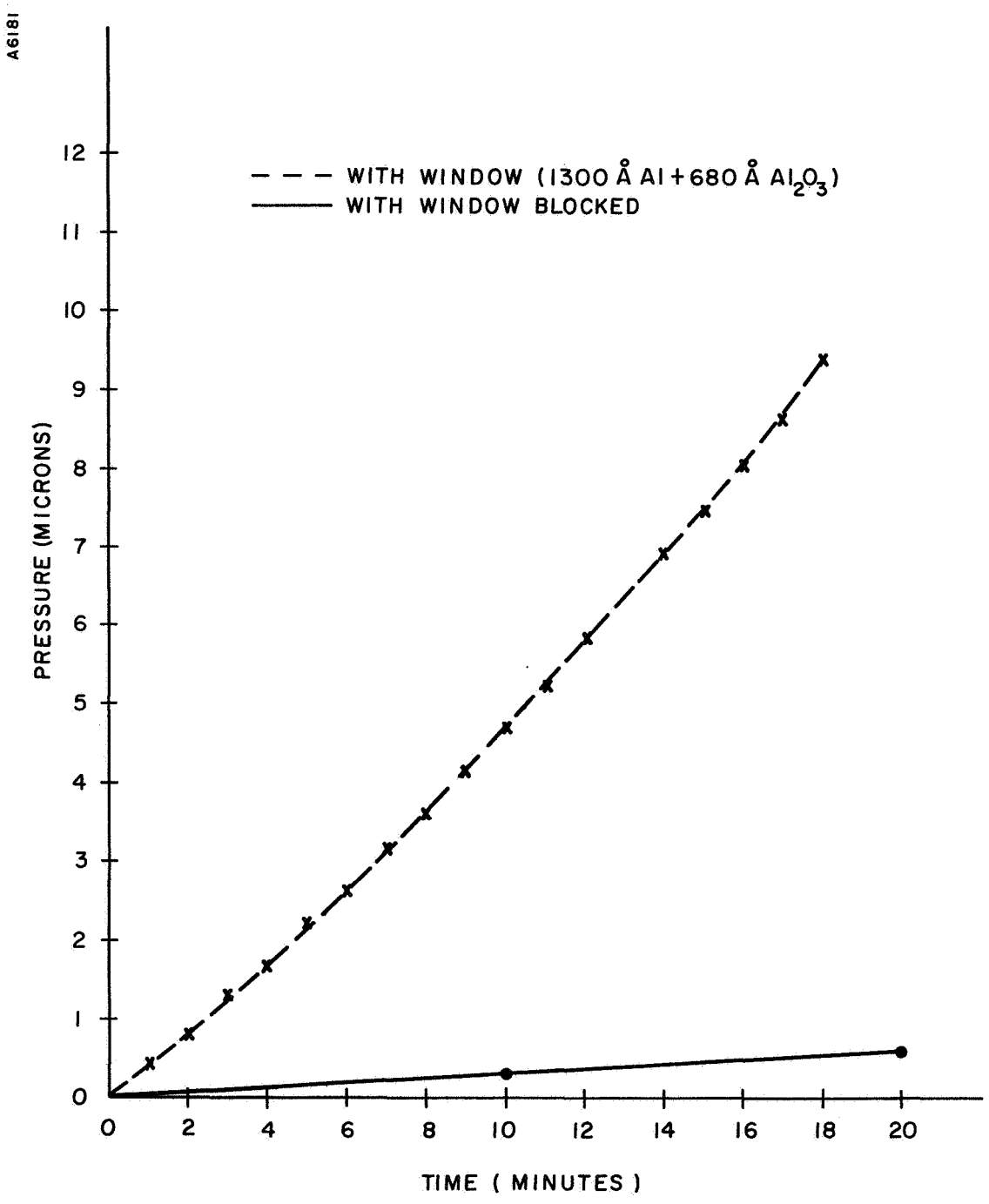


FIGURE 8. PRESSURE VERSUS TIME

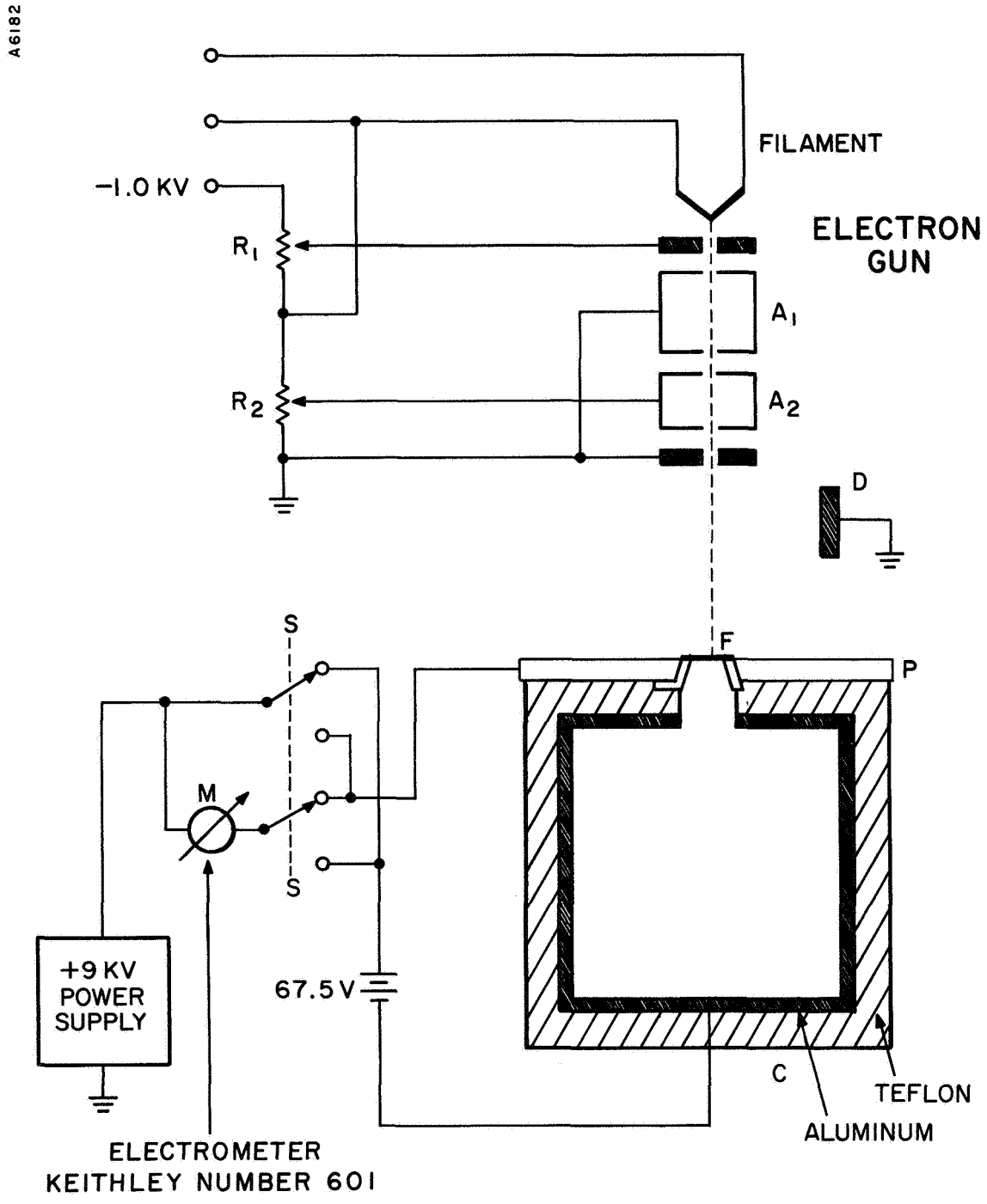


FIGURE 9. ELECTRON TRANSMISSION ARRANGEMENT

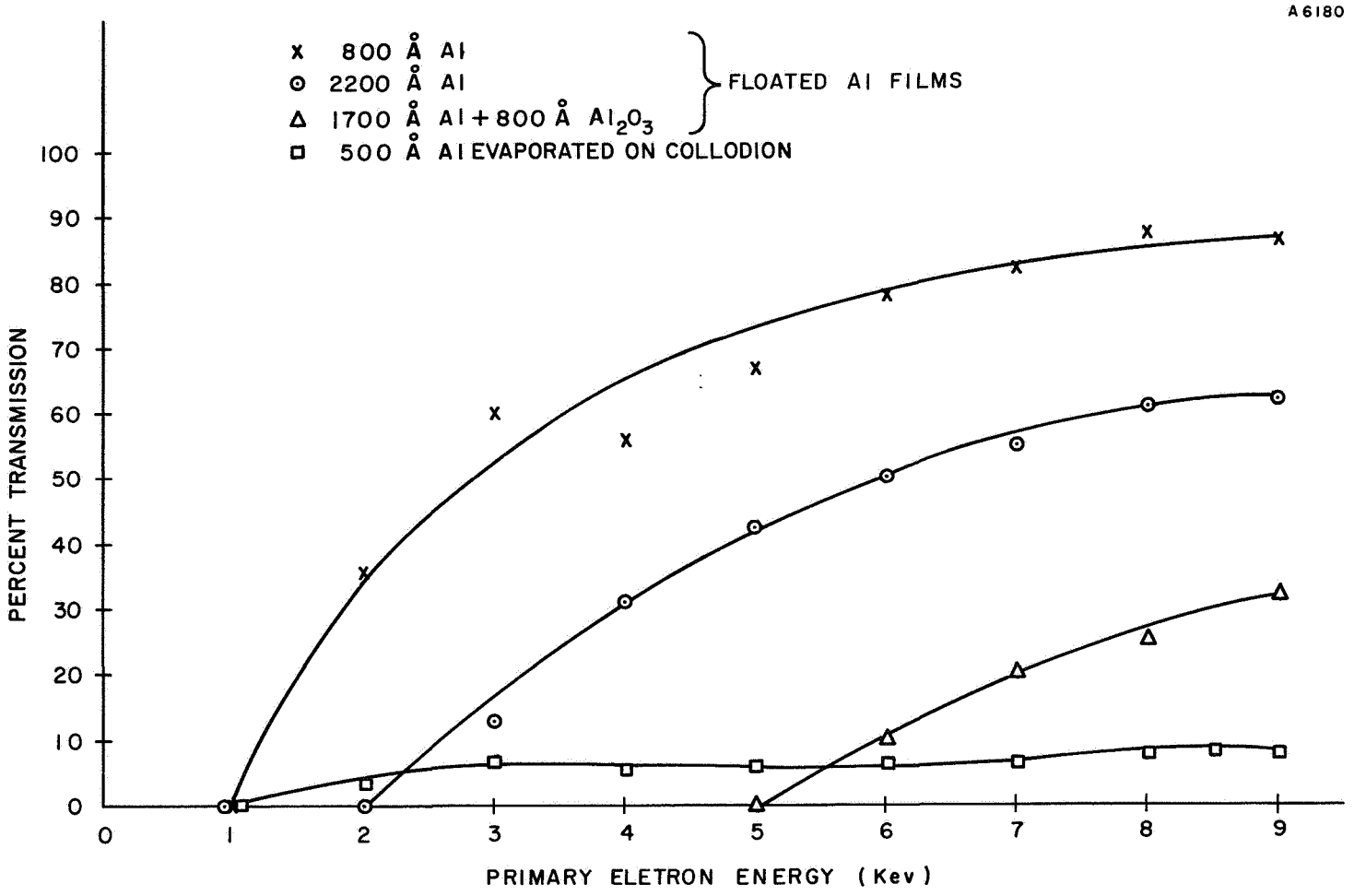


FIGURE 10. TRANSMISSION VERSUS INCIDENT ENERGY FOR Al_2O_3-Al FILMS OF DIFFERENT THICKNESSES

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TABLE 1

INVERSE STOPPING POWER AND RANGE OF ELECTRONS THROUGH ALUMINUM OXIDE FILM (THEORETICAL)

Incident Energy E (in Kev)	$(\frac{dE}{dX})^{-1}$ (in mg/cm^2 per Kev)	Range (in μ)
1	0.561×10^{-2}	0.017
2	0.977×10^{-2}	0.049
3	1.361×10^{-2}	0.094
4	1.728×10^{-2}	0.152
5	2.083×10^{-2}	0.221
6	2.429×10^{-2}	0.303
7	2.767×10^{-2}	0.396
8	3.100×10^{-2}	0.500
9	3.427×10^{-2}	0.616
10	3.750×10^{-2}	0.741
11	4.069×10^{-2}	0.878
12	4.385×10^{-2}	1.024
13	4.697×10^{-2}	1.184
14	5.007×10^{-2}	1.353
15	5.314×10^{-2}	1.530

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